Optimizing Lanthanum Chromite Interconnects for Solid Oxide Fuel Cells

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ABSTRACT

Optimizing the composition of strontium substituted lanthanum chromites to minimize expansion is not straightforward and must be done carefully to avoid compromising sinterability, electrical conductivity, and mechanical strength. In this study, a variety of lanthanum strontium chromites were modified with V, Cu, and Mg to allow densification in air at 1500°C, and their materials properties were evaluated. Both additives (• 5 mole % V and 2 mole% Cu) resulted in full densification of Sr substituted (10 and 15 mole%) lanthanum chromite. Of the compositions evaluated, only $La_{0.85}Sr_{0.15}Cr_{0.95}V_{0.05}O_3$ appeared to be a promising interconnect material, showing <0.1% expansion at 10^{-16} atm. While the coefficient of thermal expansion was less than that of stabilized zirconia, small additions of Co (1 mole%) increased the CTE to 10.1 ppm °C^{-1}. The results of this study indicated that Cu acted as an acceptor when added to lanthanum chromite, increasing both the electrical conductivity and expansion upon reduction. The effect of vanadium additions were two-fold. First, it acted as a donor in the chromite lattice effectively reducing the overall acceptor concentration. Secondly, during sintering, V reacted with Sr to form $Sr_3(VO_4)_2$, an effective sintering aid for lanthanum chromite, and further reduced the acceptor level by removing Sr from the perovskite lattice.

INTRODUCTION

The interconnect is a critical component in a planar solid oxide fuel cell (SOFC). The role of the interconnect is two-fold; it provides electrical connection between individual cells and acts as a gas separation barrier to prevent mixing of the fuel and air. The chosen material must have high electronic conductivity, low ionic conductivity, a thermal expansion match to the other cell components, good mechanical strength, and chemical and physical stability in a dual environment (air on one side, fuel on the other) at 1000°C. Typically, acceptor substituted lanthanum chromite has been the material of choice for a high-temperature ceramic interconnect.

Charge compensation for acceptor doping of lanthanum chromite is primarily electronic in oxidizing conditions (through the transition of Cr^{3+} to Cr^{4+}) (1). Under sufficiently reducing conditions, ionic charge compensation becomes more significant (through the formation of oxygen vacancies as the material becomes oxygen deficient).

The reduction of Cr and formation of oxygen vacancies cause the rare earth chromite to dilate or expand. This defect-induced dilation has been well documented (2-7) and is one of the reasons for the limited use of chromite based interconnects in planar SOFCs. It can be minimized by simply reducing the amount of Cr^{4+} (i.e., acceptor concentration) in the composition. Reducing the amount of acceptor doping, however, also results in a decrease in electronic conductivity. Therefore, achieving an optimized composition is a trade-off between minimizing the dilation and achieving a high electronic conductivity.

It has been difficult at best to optimize the composition of lanthanum chromite to achieve the desired electrical and thermal properties while minimizing expansion. Recent studies of the mechanical properties of lanthanum chromite indicate that only Sr substituted lanthanum chromite has adequate mechanical strength after exposure to a reducing environment (8). In addition, the use of strontium rather than calcium results in lower expansion in reducing environments (2,5). Other studies (2,6,7) have indicated that the additions of transition metals to the B-site are beneficial in reducing the expansion in reducing environments. Larson et al. (6) showed that addition of a donor such as vanadium on the B-site aided densification and decreased the expansion upon reduction. Their study indicated that double doping with both Mg and V resulted in lower expansions; however, the coefficient of thermal expansion and electrical conductivity of these materials did not meet the requisite requirements for an interconnect. Similar results with respect to electrical conductivity and thermal expansion were achieved in a separate study at SOFCo (7).

In order to determine which chromite compositions have the requisite minimal expansion, boundary conditions (oxygen partial pressure $P(O_2)$ and permissible expansion) must be chosen. The equilibrium oxygen partial pressure of the fuel is largely a function of the type of reforming process utilized. For example, it has been reported that the $P(O_2)$ for steam reformed methane is 10^{-16} atm (9). In this study, the partial pressure of reformed logistic fuels, such as JP8 or diesel, was evaluated. Both steam reforming and autothermal reforming were considered. For steam reformed JP8 the equilibrium (PO_2) was • 10^{-16} atm and for autothermal reforming of the same fuel, the $P(O_2)$ was $3x10^{-15}$ atm. For some applications the U. S. Department of Energy is considering reformed coal gas. Should reformed coal gas be used the $P(O_2)$ would be • 10^{-17} atm (10).

The maximum tolerable expansion is still not known and will probably vary for different SOFC designs. However, Larson et al. (6) have set the upper limit at $\delta l/l<0.05\%$, while Yasuda and Hishinuma (11) have used $\delta l/l<0.10\%$ to avoid failure of their interconnects plates. Hartvigsen et al. (7) reported both higher open circuit values and better stack performance when interconnects with expansions less than 0.10% were used. For the purposes of this study $\delta l/l=0.10\%$ was used as the upper limit of tolerable expansion and the lower boundary for $P(O_2)$ was 10^{-16} atm.

The purpose of this study was to optimize the composition of Sr-substituted lanthanum chromite to ensure that it sinters to full density at 1500°C (2 hr soak in air), has an electrical conductivity >20 S/cm at 1000°C in air and >5 S/cm at 1000°C at 10^{-16} atm $P(O_2)$, has a thermal expansion match to fully stabilized zirconia, and has minimal expansion in reducing environments. This study sought to maximize the acceptor concentration in the material while optimizing these properties.

EXPERIMENTAL PROCEDURE

All powders for this study were prepared by combustion synthesis using metal nitrate precursors and glycine as the fuel. The solutions of metal nitrates and glycine were heated on a hot plate until enough water was removed to initiate self-sustaining combustion. The ash produced by the combustion event was passed through a 100 mesh screen to break up large agglomerates, and then calcined at 1000°C for 1 hour in air. The calcined powders were uniaxially pressed (55MPa) into pellets (22 mm dia.), and then isostatically pressed at 138 MPa. The pressed compacts were sintered for 2 hours in air (1400 to 1600°C) and bulk densities of the sintered samples were determined by the Archimedes method using ethanol.

The phase development and lattice parameters of the as-synthesized and sintered materials were determined by X-ray diffraction. Thermal expansion measurements were carried out using a vertical pushrod dilatometer and fully dense samples (• 25 mm in length). The linear expansion was measured to 1200°C at 2°C/min in air. Additional dilatometric measurements were used to determine the stability of synthesized chromites in highly reducing environments. These experiments were carried out at a fixed temperature using sintered bars (• 30 mm x 3 mm x 3 mm). The $(P(O_2))$ was varied using a buffered $CO_2/Ar/H_2$ gas system. The buffered gas system allowed measurements to be made over a $P(O_2)$ range from 10^{-5} to 10^{-18} atmospheres at 1000°C.

Electrical conductivity of fully dense sintered bars (• 30 x 2 x2 mm) was measured in air as a function of temperature up to 1000°C by a four-point dc method using platinum electrodes and a 1 hour equilibration period at each temperature. The same samples were used to determine the conductivity of the chromites in a (PO_2) of 10^{-16} atm. The buffered $CO_2/Ar/H_2$ gas system was used to control the oxygen partial pressure.

RESULTS

In this paper an abbreviated notation will be used when referring to lanthanum chromite compositions. For example, $La_{0.85}Sr_{0.15}Cr_{0.95}V_{0.05}O_3$ will be referred to as LSCV-1505, where the 15 and 05 indicate the molar concentrations of Sr and V respectively, and $La_{0.9}Sr_{0.1}Cr_{093}Mg_{0.05}Cu_{0.02}O_3$ will be referred to as LSCMCu-100502, where the 10, 05, and 02 indicate the molar concentrations of Sr, Mg, and Cu.

Cu and V were chosen as dopants to lower the sintering temperature of lanthanum chromite based on the melting point of their respective oxides and their use in previous studies (6,12,13). Hayashi et al. (13) demonstrated that Cu additions to undoped lanthanum chromite resulted in enhanced densification, possibly due to the formation of a low melting lanthanum cuprate. Larson et al. (6) has shown that V additions enhance sintering through the formation of $Sr_3(VO_4)_2$ (mp 1545°C). Strontium vanadate is thought to interact with other constituent elements in the chromite to promote liquid phase sintering.

In this study, sintered samples were examined by XRD after sintering to determine the phases present. All samples containing Cu and V were phase pure (100% perovskite) after sintering to full density except LSCV-1505. This material contained • 3.5 wt% of

$Sr_3(VO_4)_2$ as a second phase. Quantitative analysis and mass balance calculations were carried out on this sample to determine the precise amount of $Sr_3(VO_4)_2$ and to estimate the composition of the lanthanum chromite after sintering. This analysis suggests that the composition of LSCV-1505 is $(La_{0.89}Sr_{0.11})_{0.98}Cr_{0.98}V_{0.02}O_3$ after sintering. The reaction mechanism for the formation of $Sr_3(VO_4)_2$ and densification in this chromite have been described elsewhere (14). The present data, however, indicates that V has a limited solubility in the perovskite (LSC) lattice. In addition, it indicates that the perovsktie has a 27% reduction in its acceptor content, through the formation of $SrCrO_4$ and reaction with V to form $Sr_3(VO_4)_2$. This implies that LSCV-1505 will have a significantly lower expansion during reduction. The fact that all other samples were phase pure implies that the V solubility may be a function of the Sr concentration for these chromites. In the work of Larson (6), samples similarly doped with Mg, Sr, and V were prepared by the glycine-nitrate synthesis method. In that study, the samples contained $Sr_3(VO_4)_2$ after sintering, in contrast to the samples in this study, which were phase pure. The reason for the discrepancy is uncertain. It may have resulted from differences in the synthesis, calcination, or sintering conditions.

The densification behavior of lanthanum chromite containing 10 and 15 mole% Sr with Cu or V is shown in Figure 1. The data indicates that both V and Cu are effective sintering aids for LSC, and that densities greater than 95% of theoretical were achieved for all four compositions at 1500°C. This is a significant improvement over undoped LSC-15 and LSC-10, and indicates that V additions are an effective sintering aid.

Figure 2 shows the densification data for samples doped with both Sr and Mg such that the total acceptor level of these two dopants was equal to 15 mole percent. In addition, these samples were further modified with Cu or V to improve their sinterability. Figure 2 indicates that both Cu and V are beneficial sintering aids significantly lowering the sintering temperature of these materials. Densities of samples with V additions are similar to those prepared by Larson (6). A further comparison of the two data sets indicates that additions of Cu as low as 2 mole% improve the density of sintered materials. While Cu is an effective sintering aid, the role it plays in sintering is unknown at present.

Linear thermal expansion coefficients (TEC) were measured on sintered specimens from room temperature to 1200°C (Table I). Vanadium doping has little affect on the TEC, while Cu slightly decreased the TEC. Of the samples tested only LSCV-1505 and LSC-15 had thermal expansion coefficients close to that of yttria stabilized zirconia ($\alpha=10.5 \times 10^{-6} °C^{-1}$). The phase transition temperature was obtained from the dilatometry traces. The orthorhombic to rhombohedral phase transition was less than room temperature (27°C) for samples containing 15 mole % Sr. For samples with less than 15 mole% Sr, except for LSCV-1005, a phase transition was observed. It is well known that additions of Sr to lanthanum chromite result in a decrease in the transition temperature (14). It has also been shown that Mg additions increase the transition temperature (6). The affect of Cu and V on the phase transition temperature was determined by comparing the transition temperature of LSCV-1005 and LSCCu-1005 to LSC-10. This comparison indicated that Cu increased the transition temperature while V reduced the transition temperature.

Table I.   Coefficient of thermal expansion (25°C to 1200°C) and orthorhombic to rhombohedral phase transition temperature for selected lanthanum chromites.

Composition	Thermal Expansion Coefficient $(10^{-6} \, °C^{-1})$	Phase Transition Temperature (°C)
$La_{0.9}Sr_{0.5}CrO_3$	10.05	57
$La_{0.85}Sr_{0.15}CrO_3$	10.52	<25
$La_{0.87}Sr_{0.1}Cr_{0.95}Cu_{0.05}O_3$	9.81	54
$La_{0.85}Sr_{0.15}Cr_{0.98}Cu_{0.02}O_3$	9.91	<25
$La_{0.9}Sr_{0.1}Cr_{093}Mg_{0.05}Cu_{0.02}O_3$	9.61	60
$La_{0.95}Sr_{0.05}Cr_{088}Mg_{0.1}Cu_{0.02}O_3$	9.42	150
$La_{0.9}Sr_{0.1}Cr_{0.95}V_{0.05}O_3$	9.87	<25
$La_{0.85}Sr_{0.15}Cr_{0.95}V_{0.05}O_3$	10.00	<25
$La_{0.9}Sr_{0.1}Cr_{09}Mg_{0.05}V_{0.05}O_3$	9.64	89
$La_{0.95}Sr_{0.05}Cr_{085}Mg_{0.1}V_{0.05}O_3$	9.22	205

Plots of the electrical conductivity measurements are not shown in this paper, however, the data for the LSC compositions are consistent with small polaron conduction (i.e., localized electronic charge carriers having a thermally activated mobility). The data indicate that the conductivity of Cu doped samples increased significantly over the LSC-15, indicating that Cu acts as an acceptor in the perovskite lattice. It will be noted, however, that the increase in conductivity resulting from just 2 mole% additions of Cu is much greater than can be explained by simply increasing the total acceptor content from 0.15 (LSC-15) to 0.17 (LSCCu-1502). It appears that the presence of a small amount of Cu provides another undetermined mechanism for enhancing the conductivity.

The electrical conductivity of LSCV-1505 is similar to that of LSC-15 (Table II). However, LSC-15 is not fully dense and it would be expected that it would have a higher conductivity if it were dense. This would lead to the conclusion that V is a donor as suggested by earlier studies (6). The electrical conductivity (1000°C, air) of all the samples evaluated in this study are tabulated in Table II. The conductivity generally increases with increasing acceptor concentration for samples measured at 1000°C in air. The electrical conductivity of the samples tested were higher than those of previous studies evaluating the stability and properties of chromites in a reducing environment (7). This is due to the higher acceptor levels in the chromites evaluated in this study.

Table II also shows the electrical conductivity for the same chromites after equilibration at 10^{-16} atm $P(O_2)$. The conductivity decreases in all cases, with the smallest decrease being observed for LSCV-1050 and LSCV-1505. This data also indicates that as the acceptor dopant was shifted from the A-site (Sr) to the B-site (Mg), larger decreases in the conductivity were measured upon reduction. This is contrary to the data of Hartvigsen et al. (7) for samples modified only with Mg. This implies that there is an interdependence between the dopant distribution or site occupancy.

The isothermal linear expansion of sintered LSC compositions at 1000°C are shown as a function of ambient $P(O_2)$ in Figure 3. The expansions shown are referenced to the

initial sample length (at 1000°C) in CO_2. Due to the thermodynamic stability of these chromites toward reduction, no significant expansion occurred until the $P(O_2)$ was

Table II. Electrical conductivity (S/cm) in air and at 10^{-16} atm $P(O_2)$ for selected lanthanum chromite compositions.

Composition	Conductivity (S/cm, 1000°C, air)	Conductivity (S/cm, 1000°C 10^{-16} atm $P(O)_2$)
$La_{0.9}Sr_{0.5}CrO_3$	----	----
$La_{0.85}Sr_{0.15}CrO_3$	21.8	11.5
$La_{0.87}Sr_{0.1}Cr_{0.95}Cu_{0.05}O_3$	23.9	6.2
$La_{0.85}Sr_{0.15}Cr_{0.98}Cu_{0.02}O_3$	32.5	9.9
$La_{0.9}Sr_{0.1}Cr_{093}Mg_{0.05}Cu_{0.02}O_3$	----	----
$La_{0.95}Sr_{0.05}Cr_{088}Mg_{0.1}Cu_{0.02}O_3$	----	----
$La_{0.9}Sr_{0.1}Cr_{0.95}V_{0.05}O_3$	9.7	8.5
$La_{0.85}Sr_{0.15}Cr_{0.95}V_{0.05}O_3$	19.9	15.4
$La_{0.9}Sr_{0.1}Cr_{09}Mg_{0.05}V_{0.05}O_3$	15.2	8.2
$La_{0.95}Sr_{0.05}Cr_{085}Mg_{0.1}V_{0.05}O_3$	12.9	4.8

reduced to 10^{-10} atm or lower. The expansion in these samples increased with decreasing $P(O_2)$ and with increasing acceptor content. These trends are consistent with those previously reported for doped lanthanum chromite (2-5).

It has been proven that the expansion in these materials is due the reduction of Cr^{4+} in the reducing environments and the related change in size of the Cr ion (2,6). It has also been shown that donor substitution into the lattice reduces the expansion by reducing the overall acceptor concentration (2,6). The data in these figures show that the measured expansions of V substituted chromites are less than Cu modified chromites, as expected for donor and acceptor substituted materials. This is consistent with the findings showing a decrease in the acceptor concentration due to removal and reaction of Sr with V in LSCV-1505. The expansion upon reduction in all of the materials studied was proportional to the initial acceptor concentration following:

$$[Cr_{Cr}^{\bullet}] = ([Sr_{La}^{'}] + [Mg_{Cr}^{'}] - [V_{Cr}^{\bullet}] + [Cu_{Cr}^{'}]) - 2[V_O^{\bullet\bullet}]$$ [1]

Discussion

Based on the samples tested and the collected data, only $La_{0.85}Sr_{0.15}Cr_{0.95}V_{0.05}O_3$ and $La_{0.9}Sr_{0.1}Cr_{09}Mg_{0.05}V_{0.05}O_3$ have a minimal expansion under reducing conditions and an electrical conductivity greater than 20 S/cm at 1000°C in air and greater than 5 S/cm at 1000°C in 10^{-16} atm $P(O)_2$. However, the coefficient of thermal expansion of each materials does not match that of fully stabilized zirconia. This can easily be resolved by adding small amounts (<5 mole%) of Co to the composition. Cobalt additions have been shown to increase the thermal expansion of many complex perovskite materials including

lanthanum chromite (15). In addition, Co additions effectively reduce the phase transition temperature (14).

Co was added (1 and 3 mole%) to both $La_{0.85}Sr_{0.15}Cr_{0.95}V_{0.05}O_3$ and $La_{0.9}Sr_{0.1}Cr_{09}Mg_{0.05}V_{0.05}O_3$ such that the new compositions were $La_{0.85}Sr_{0.15}Cr_{0.94}V_{0.05}Co_{0.01}O_3$ and $La_{0.9}Sr_{0.1}Cr_{0.87}Mg_{0.05}V_{0.05}Co_{0.03}O_3$, respectively. The materials properties of these modified chromites were measured and are shown in Table III. Co additions to LSCV-1505 resulted in a slightly lower electrical conductivity in air while the conductivity of LSCMV-100505 was higher. Both compositions have an electrical conductivity greater than 5 S/cm in reducing environments. Additions of Co are generally an effective method to increase the electrical conductivity of lanthanum chromite in air. Previous studies (2) have shown that Co is in the 3+ oxidation state in the perovskite lattice. As such, it does not act as an acceptor dopant and should not enhance the conductivity when present in small quantities. When present in larger quantities, it substantially increases the conductivity of perovskites, which is not surprising given the very high conductivity of doped lanthanum cobaltite.

Table III. Electrical conductivity (S/cm; in air and at 10^{-16} atm $P(O)_2$) and coefficient of thermal expansion lanthanum strontium chromites modified with Co.

Composition	Thermal Expansion Coefficient $(10^{-6}\ °C^{-1})$	Conductivity (S/cm, 1000°C, air)	Conductivity (S/cm, 1000°C 10^{-16} atm $P(O_2)$
$La_{0.87}Sr_{0.1}Cr_{0.935}Cu_{0.05}Co_{0.015}O_3$	10.4	25.6	----
$La_{0.85}Sr_{0.15}Cr_{0.935}V_{0.05}Co_{0.015}O_3$	10.5	22.9	12.5
$La_{0.9}Sr_{0.1}Cr_{0.92}V_{0.05}Co_{0.03}O_3$	10.4	12.8	7.8

CONCLUSIONS

The results of this study indicate that chromite compositions with • 10-12 mole acceptor can be developed that: 1) sinter to full density at 1500°C (2hr in air), 2) have an electrical conductivity >20 S/cm at 1000°C in air, 3) have an electrical conductivity >5 S/cm (1000°C at 10^{-16} atm $P(O_2)$), 4) have a thermal expansion match to fully stabilized zirconia, and 5) have a minimal expansion in reducing environments (≤0.1%).

This study showed that samples can be fully densified by modifying the chromite compositions with 5 mole% V or 2 mole % Cu. Vanadium has a limited solubility in the LSC lattice and upon sintering it reacts with $SrCrO_4$ to form $Sr_3(VO_4)_2$, an effective sintering aid. This reaction reduces the total acceptor concentration in the chromite by the removal and reaction of Sr with V, thus reducing the maximum expansion of the material in a reducing environment. While the TECs of most the compositions prepared and tested were lower than that of fully stabilized zirconia, all could be raised by the addition of Co.

The electrical conductivity of the samples developed in this work reflect higher acceptor levels than in samples developed in previous work. Additionally, this study

showed that samples with relatively high acceptor concentrations could be developed to meet all requirements of a solid oxide fuel cell interconnect. While the mechanical properties of these materials are unknown at present, they are being evaluated and will be reported at a later date.

ACKNOWLEGEMENTS

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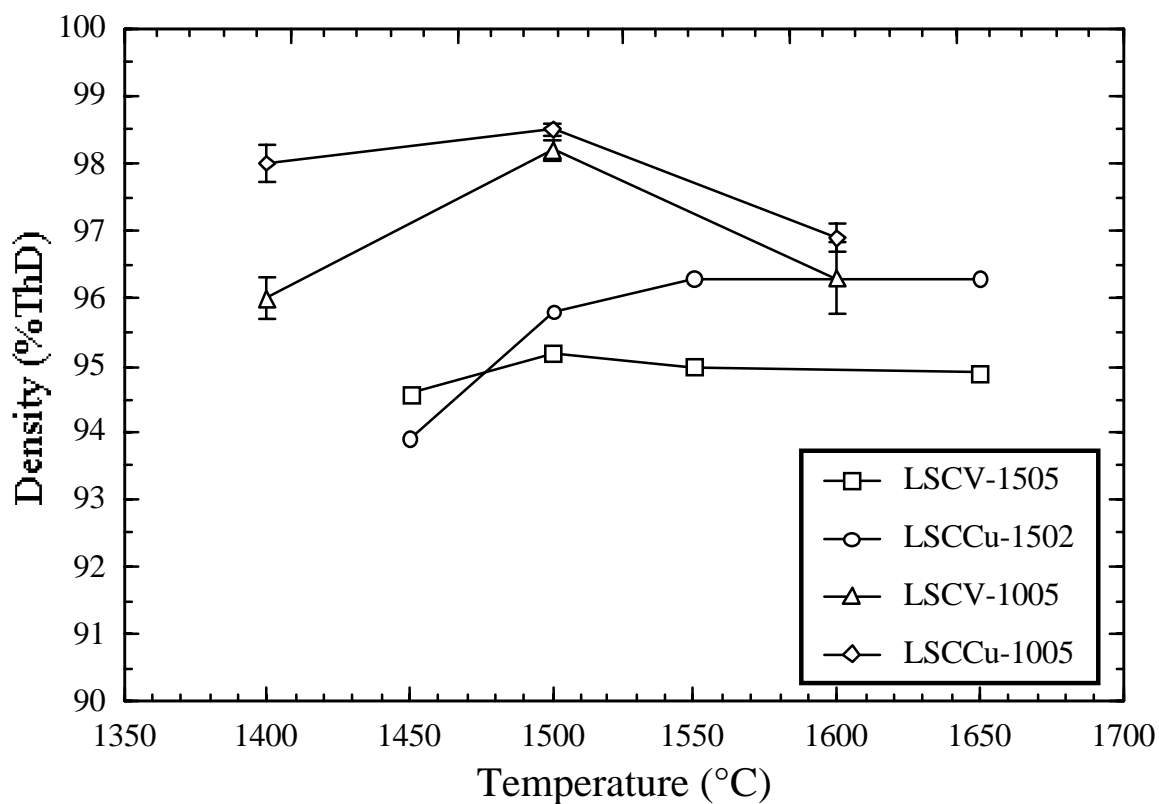
Figure 1. Theoretical density as a function of temperature for LSCV-1505, LSCV-1005, LSCCu-1502, and LSCV-1005.
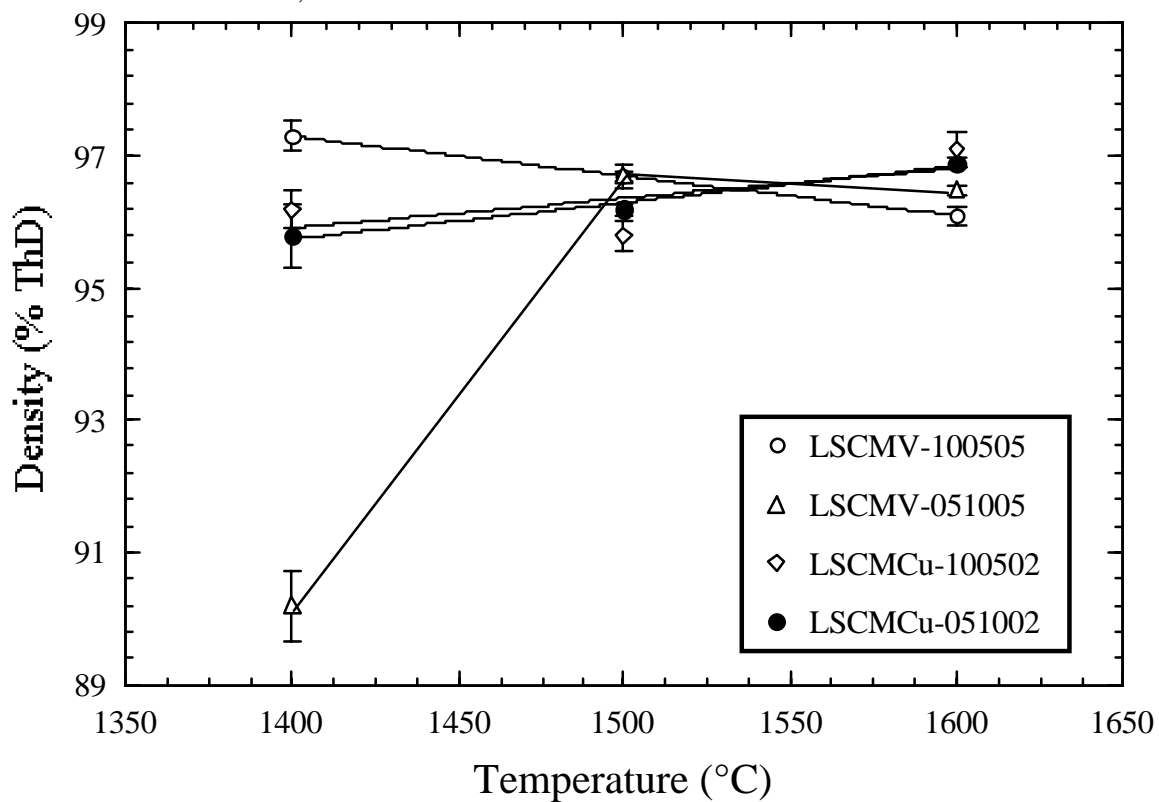

Figure 2. Theoretical density as a function of temperature for Sr-substituted lanthanum chromites containing Mg with Cu or V as sintering aids.
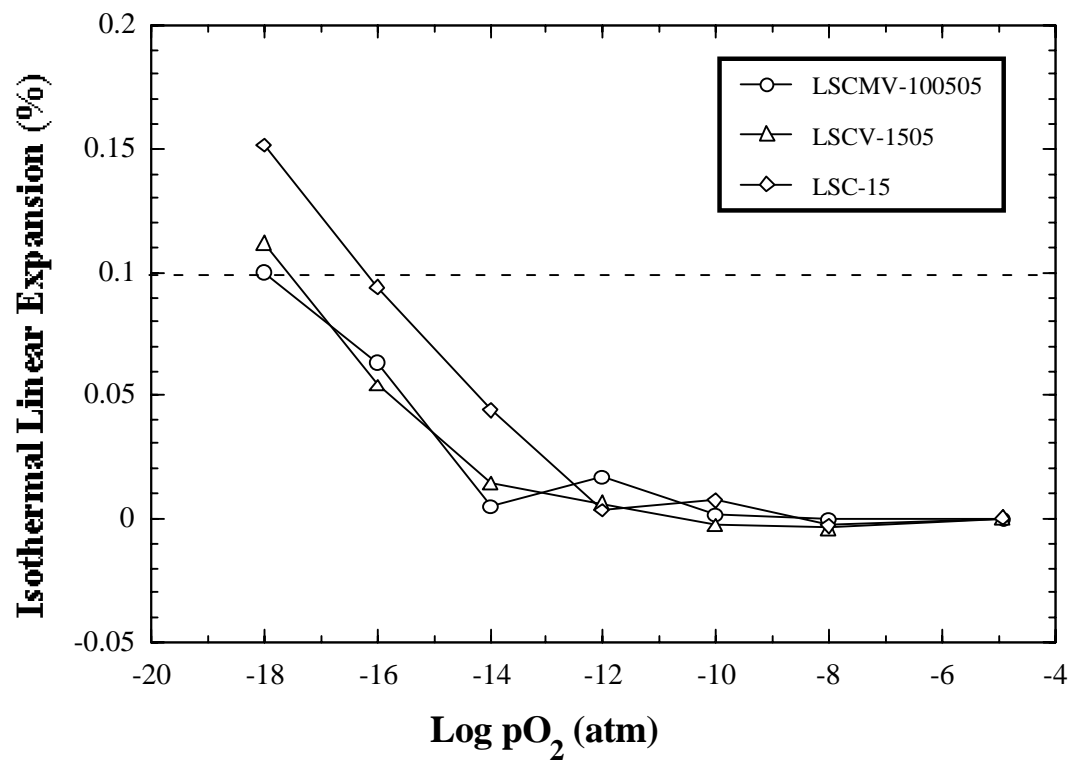
Figure 3. Isothermal Expansion of LSCV-1505 and LSCMV-105050 as a function of oxygen partial pressure at 1000°C.